

Avoiding preamorphization damage in MeV heavy ion-implanted silicon

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Implantation of 1.0 MeV ^{115}In in Si results in secondary-defect formation during subsequent 900 °C annealing if the total number of displaced Si atoms is greater than $1.6 \times 10^{17}/\text{cm}^2$, achieved with a dose near $1.5 \times 10^{13}/\text{cm}^2$. We demonstrate, though, that higher total In doses can be introduced without forming secondary defects by repetitive subthreshold implants each followed by an anneal to remove the implant damage. While a single $6 \times 10^{13} \text{ In}/\text{cm}^2$ implant results in a high density of dislocation loops after annealing, instead using four separate $1.5 \times 10^{13} \text{ In}/\text{cm}^2$ implants each followed by an anneal leads to the formation of only a few partial dislocations.

Ion implantation in crystals at or below room temperature with doses below the amorphization threshold creates point defects and simple defect clusters. During subsequent thermal annealing these simple defects may agglomerate and eventually form extended, or secondary, defects. For example, if there are enough interstitials, they will condense to form interstitial platelets which lead to dislocation loops.^{1,2} Work on MeV implants of B, P, and As has suggested that there is a critical implant dose, $\approx 2 \times 10^{13} - 1 \times 10^{14}/\text{cm}^2$, above which secondary defects will form.³⁻⁵ In recent work,⁶ however, we have determined that B implants between 50 keV and 2 MeV result in secondary-defect formation if and only if the total number of displaced Si atoms exceeds a critical value. Since the B dose required to displace this critical number of Si atoms changes by an order of magnitude over the energy range investigated, the important parameter is not the dose, but instead the critical number of displaced Si atoms, N_c .

We have found that for a single ^{115}In implant the critical number of displaced silicon atoms is $N_c \approx 1.6 \times 10^{17}/\text{cm}^2$, which is achieved by a $1.5 \times 10^{13}/\text{cm}^2$ 1.0 MeV In implant.⁶ A lower dose In implant does not lead to secondary-defect formation. Here, it is shown that formation of secondary defects can be avoided for even higher total In doses by using repetitive subthreshold implants each followed by an anneal to remove the accumulated damage. This demonstrates that the critical parameter for secondary-defect formation is the damage in the crystal and not the implanted dose. A single implant of $6 \times 10^{13} \text{ In}/\text{cm}^2$ results in a high concentration of dislocation loops after a 15 min anneal at 900 °C. However, if the total dose is instead implanted in four $1.5 \times 10^{13} \text{ In}/\text{cm}^2$ steps each followed by a 900 °C anneal, only a very low density of elongated partial dislocations results. Knowledge of N_c thus leads to the ability to engineer the final defect structure.

Implants of 1.0 MeV ^{115}In into Si(100) were performed at room temperature. The total In current on target ($1.5 \times 1.5 \text{ cm}^2$) was kept below 10 nA to avoid beam heating. Anneals were performed in a vacuum furnace (base pressure $\approx 1 \times 10^{-7}$ Torr) at 900 °C. The damage in the as-implanted and annealed samples was measured using Rutherford backscattering spectrometry (RBS) in the channeling configuration with 2.0 MeV He. Secondary-defect structures were investigated with cross-sectional trans-

mission electron microscopy (XTEM).

Four samples were implanted to the same total dose of $6 \times 10^{13} \text{ In}/\text{cm}^2$ using various implant/anneal cycles as listed in Table I. Samples 1 and 2 were made with a single $6 \times 10^{13} \text{ In}/\text{cm}^2$ implant followed by 900 °C anneals for 15 and 60 min, respectively. Sample 3 was formed with two $3 \times 10^{13} \text{ In}/\text{cm}^2$ implants each followed by 15 min, 900 °C anneal. Finally, sample 4 received a series of four $1.5 \times 10^{13} \text{ In}/\text{cm}^2$ implants each followed by 15 min, 900 °C anneal. RBS confirmed that all samples were implanted with the same total In dose.

The RBS/channeling spectra in Fig. 1 show the damage in the samples after each In implant. For all implants the dechanneling maximum (the damage peak) occurs at a depth of 350 nm, somewhat shallower than the In range of 410 nm measured by RBS. Analyzing the dechanneling using the method of Chu *et al.*⁷ yields the number of displaced Si atoms for each implant, as listed in Table I. The damage produced by a single In implant is nonlinear versus dose, with $6 \times 10^{13} \text{ In}/\text{cm}^2$ introducing $\approx 7 \times$ the damage of a $1.5 \times 10^{13} \text{ In}/\text{cm}^2$ implant. For the multiple implant/anneal samples, the implants into annealed samples lead to dechanneling levels that are essentially identical to those of the first implant. The damage profile for sample 4 that is slightly lower than the rest is the third out of the four.

RBS/channeling measurements (not shown) for each sample following the final anneal illustrate an increase in dechanneling for samples 1, 2, and 3 at a depth of 350 nm, indicative of secondary defects near the peak of the as-implanted damage profile. However, the channeling spectrum for sample 4 [$4 \times (1.5 \times 10^{13} \text{ In}/\text{cm}^2 + 15 \text{ min at } 900^\circ\text{C})$] is indistinguishable from the virgin Si(100) spectrum. Thus the density of any secondary defects in sample 4 is considerably lower than in sample 1, 2, or 3.

The XTEM micrographs in Fig. 2 confirm this. Both samples 1 and 2 contain a band of dislocation loops with average diameter $\bar{\phi} = 15 \text{ nm}$ centered on the damage profile peak at 350 nm [Figs. 2(a) and 2(b)]. The first implant/anneal cycle for sample 3 ($3 \times 10^{13} \text{ In}/\text{cm}^2 + 15 \text{ min at } 900^\circ\text{C}$ anneal) also results in a band of dislocation loops, of similar size but lower density [Fig. 2(c)]. After the second implant/anneal sequence, the average size of these loops has increased from $\bar{\phi} \approx 10 \text{ nm}$ to $\bar{\phi} \approx 35 \text{ nm}$ but the number of loops has not changed significantly [Fig.

TABLE I. Implant and anneal procedures used to prepare the samples along with the number of Si atoms displaced by a single implant in the series and the total number of Si atoms displaced by all implants to make the sample.

Sample number	Indium implant and anneal schedule	Displaced silicon atoms per implant prior to annealing (/cm ²)	Total silicon atoms displaced by all implants (/cm ²)
1	6×10^{13} In/cm ² , 15 min at 900 °C	1.1×10^{18}	1.1×10^{18}
2	6×10^{13} In/cm ² , 60 min at 900 °C	1.1×10^{18}	1.1×10^{18}
3	$2 \times (3 \times 10^{13})$ In/cm ² , 15 min at 900 °C	3.7×10^{17}	7.4×10^{17}
4	$4 \times (1.5 \times 10^{13})$ In/cm ² , 15 min at 900 °C	1.6×10^{17}	6.4×10^{17}

2(d)]. Sample 4's first implant and anneal results in no identifiable defects [Fig. 2(e)]. Even after all four implant/anneal cycles, only a very low concentration of partial dislocations can be observed in XTEM [Fig. 2(f)]. The secondary-defect density is significantly lower in sample 4 than in the other samples.

A single 6×10^{13} In/cm² implant (samples 1 and 2) displaces 1.1×10^{18} Si/cm², some seven times the critical number required for secondary-defect formation ($N_c \approx 1.6 \times 10^{17}$ /cm²), resulting in a high density of dislocation loops after annealing. For sample 3, the first 3×10^{13} In/cm² implant also displaces more than the critical number of Si atoms and secondary defects form during the first anneal. The second implant creates additional point defects which can be trapped by the existing dislocation loops during the following anneal. The result is a lower density of larger loops than in samples 1 or 2. It is interesting to note that the different dislocation morphologies in samples 1, 2, and 3 all lead to similar RBS dechanneling levels. In contrast, the first implant for sample 4 does not lead to secondary-defect formation. Thus, for this implant the number of displaced Si atoms is just at or below N_c . The few dislocations remaining after the final anneal are identified as partials lying on (111) planes, intermediate in the formation of the dislocation loops seen in the other samples.⁸

We believe that a 1.5×10^{13} 1.0 MeV In/cm² implant

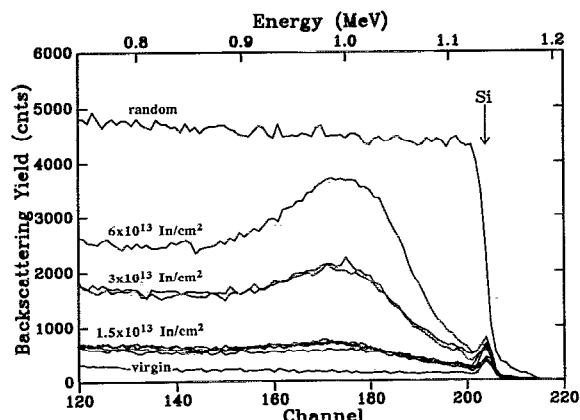


FIG. 1. Si portion of RBS/channeling spectra following In implantations. The single 6×10^{13} 1.0 MeV In/cm² implant (samples 1 and 2) leads to a high level of damage and each 3×10^{13} In/cm² implant for sample 3 results in an intermediate level of damage. For sample 4, each 1.5×10^{13} In/cm² implant only causes a low amount of damage. Random and channelled spectra of virgin Si(100) are also shown.

displaces a number of Si atoms just at or below N_c . The few partial dislocations observed in the final structure of sample 4 could be caused by one implant with slightly more damage than the others. However, the observed dislocations most likely appeared during the final anneal; otherwise, later implant damage could have enlarged these defects, as observed for sample 3, and led to loop formation. But, the lowest damage profile in the repeated series (Fig. 1) is from the third implant; thus the first implant, which shows no extended defects after annealing, introduced as much damage as the final implant. This then suggests that incomplete annealing of the largest damage clusters from one implant to the next eventually leads to dislocation formation after enough steps. It is possible that by using more steps with smaller In doses between anneals would have led to dislocation-free material. An alternative explanation for

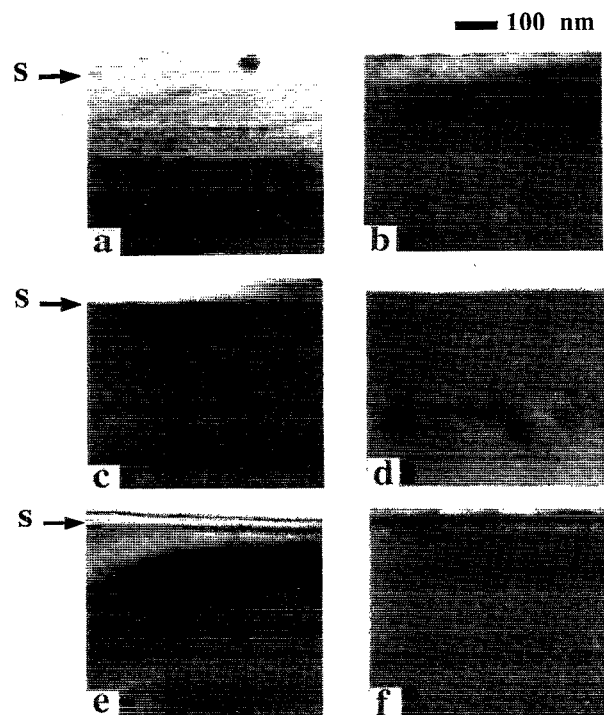


FIG. 2. Bright-field TEM micrographs along [110] following anneals of (a) sample 1 (6×10^{13} In/cm², 15 min at 900 °C), (b) sample 2 (6×10^{13} In/cm², 60 min at 900 °C), (c) sample 3 after the first implant/anneal cycle (3×10^{13} In/cm², 15 min at 900 °C), (d) sample 3 after the final cycle [$2 \times (3 \times 10^{13})$ In/cm², 15 min at 900 °C], (e) sample 4 after the first cycle (1.5×10^{13} In/cm², 15 min at 900 °C), and (f) sample 4 after the final cycle [$4 \times (1.5 \times 10^{13})$ In/cm², 15 min at 900 °C].

the formation of partial dislocations is that the In solid solubility limit has been exceeded.

We attribute the sharply reduced density of secondary defects in sample 4 to periodic annealing out of the implant-induced damage before enough has accumulated to cause dislocation formation. Annealing of single 3 and 6×10^{13} 1.0 MeV In/cm² implants led to secondary defects while annealing the first 1.5×10^{13} 1.0 MeV In/cm² implant for sample 4 resulted in dislocation-free crystal Si, demonstrating that a critical amount of damage must exist to form secondary defects. Although the total number of displaced Si atoms and the total In dose were similar in samples 3 and 4, very few extended defects formed in sample 4, again showing that the critical parameter for secondary-defect formation is the number of displaced Si atoms during a given anneal. The low density of secondary defects in sample 4 cannot be caused by the total anneal time, since sample 2 was also annealed for 60 min yet still retains a high density of dislocation loops. All samples received the same total In dose so there are no effects of different In concentration (peak concentration $\approx 4 \times 10^{18}$ /cm³ at a depth of 410 nm) on dislocation formation in the different samples. Also, the fact that the band of dislocations is centered on the damage peak and not the In profile indicates that the In has little influence on dislocation formation. Furthermore, we have also found that the critical number of displaced Si atoms for secondary-defect formation following Sb implants is the same as for In.⁶ Since Sb has nearly the same mass as In but a higher solubility,⁹ this again rules out In solubility and chemical effects on the secondary-defect formation.

Secondary defects form during annealing after implantation only if the number of displaced Si atoms exceeds a critical number N_c ; for ^{115}In $N_c \approx 1.6 \times 10^{17}$ /cm², achieved

with a dose of 1.5×10^{13} 1.0 MeV In/cm². We have demonstrated that the total In doses higher than this (here up to 6×10^{13} In/cm²) can be implanted without forming a significant number of secondary defects by periodically annealing out the subthreshold damage. Preliminary work shows similar behavior with both B, P, and As implants. These results emphasize the fact that the critical parameter for formation of secondary defects is the amount of damage present in the crystal during annealing, not the implant dose, and that secondary defects can be suppressed or eliminated by using proper implant and annealing conditions.

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¹R. S. Nelson, in *Proceedings of the International Conference on Radiation Damage and Defects in Semiconductors*, Reading, England, 1972 (Institute of Physics, London, 1973), p. 140.

²J. W. Corbett, J. P. Karins, and T. Y. Tan, *Nucl. Instrum. Methods* **182/183**, 457 (1981).

³M. Tamura, N. Natsuaki, Y. Wada, and E. Mitani, *J. Appl. Phys.* **59**, 3417 (1986).

⁴M. Tamura and N. Natsuaki, *Jpn. J. Appl. Phys.* **25**, L474 (1986).

⁵M. Tamura, N. Natsuaki, Y. Wada, and E. Mitani, *Nucl. Instrum. Methods B* **21**, 438 (1987).

⁶R. J. Schreutelkamp, J. R. Liefting, W. X. Lu, V. Raineri, J. S. Custer, F. W. Saris, B. X. Zhang, Z. L. Wang, and K. T. F. Janssen, presented at 7th International Conference on Ion Beam Modification of Materials, Knoxville, TN, 1990; B. X. Zhang, Z. L. Wang, R. J. Schreutelkamp, F. W. Saris, A. Y. Du, and Q. Li, *Nucl. Instrum. Methods B* **48**, 425 (1990).

⁷W. K. Chu, J. W. Mayer, and M.-A. Nicolet, *Backscattering Spectrometry* (Academic, New York, 1978).

⁸J. P. Hirth and J. Lothe, *Theory of Dislocations* (Wiley, New York, 1982).

⁹D. de Cogan, *Properties of Silicon* (INSPEC, New York, 1988), p. 390.